Sample Environment News

June 17, 2006

11T Magnet Now Goes to 11T...

...on BT7. BT7 has very few of the magnetic objects commonly found around the sample position at other beam lines. By hanging the magnet slightly above the goniometer with precision scales, we found that the magnet can run up to 11T without experiencing any critical forces. The 11T magnet's field is asymmetric, making it particularly useful in polarized beam experiments. Its temperature range is 1.5-300K with a regular sample stick or 0.3-300K with the ^{3}He insert. Additionally, the sample stick has a motor controlled rotation stage that is compatible with the sample rotation (angle 3) motor controllers found at all NCNR instruments.

What Makes the ARS 10K CCRs So Cool?

They have all been converted into 5K CCRs. Over the last year, the sample environment team has shipped batches of the ARS 10K CCRs for low temperature modifications.* The piston and cylinder housing were both upgraded, but all of the outer pieces, including the motor and compressor, remain the same. Now, a total of eight bottom-loading CCRs reach 5K. Note that the ARS CCRs are identified by having either blue or blue and white compressors. The Leybold CCRs with orange and black compressors will not be modified; in fact, they will slowly be cycled out of the NCNR.

Widely Anticipated "How to Refill Liquid Helium" Movie Premieres on Internet

Watch an all-star cast take the mystery out of refilling cryostats with liquid cryogens. This 6:35 minute movie is a comprehensive supplement to the short manual found in the equipment booklets. It can be used as either an introduction to or review of refilling cryostats. The visual cues and directives are intended to help users avoid common pitfalls and make the refilling process generally less cumbersome. Available in multiple formats, the movie as well as other videos on loading and unloading sample cans can be found under the "How-to Videos" subsection of the sample environment website:
http://www.ncnr.nist.gov/equipment/ancequip.html

*Certain commercial equipment, instruments, or materials are identified in this paper to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

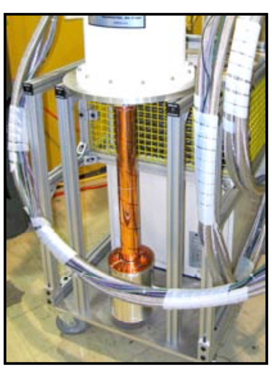
New TLCCRS Arrive!

A fresh mix of Top Loading Closed Cycle Refrigerators (TLCCRs) are filtering into the sample environment fleet. We have two short-tailed TLCCRs for general use while others with customized tail sets are dedicated to HFBS, DCS, and FANS. The noticeable asymmetry of the TLCCR is from the outer vacuum jacket enclosing the cold head to the side of the sample well.

The CCRs are able to reach temperatures as low as 5K and as high as 800K. For low temperatures, a sample is cooled by exchange gas in the sample well. To reach high temperatures, the sample well must be evacuated and a heater on the sample stick warms the sample.

Instead of designating an entire CCR for a special sample environment as in the past, the sample sticks can be modified. Pre-configured sample sticks will be available for gas-loading, high pressure, and high voltage experiments. All of these CCRs have a ballooned sample well to reduce background from the aluminum windows. The sample changing process is the same as that currently used on the orange cryostats.

We are currently characterizing the new TLCCRS, but preliminary results indicate that a sample change at base temperature takes about an hour for the sample to reach base temperature. The TLCCRs currently take 8 hours to cool to base temperature from 300K and 2.5 hours to warm from base temperature back to 300K. However, while the cryostats can reach 800K, our Aluminum sample cans and heat shields cannot. So for the moment, we must limit temperatures to 700K.



Properly Operating a High Temperature CCR

When using a high temperature CCR above 300K, always run the compressor to avoid overheating the cold head. Even after finishing an experiment, use the compressor until the CCR cools to room temperature. Because the expander contains lead, a hazardous waste clean up may be required if the cold head is severely overheated. As an extra precaution, a safety switch stops the heater if the cold head reaches 340K. To protect the cold head and to optimize PID temperature control, run the compressor for two hours before starting the first temperature set point.

CCRs and Cadmium Safety

Cadmium masks should rarely be used above room temperature because cadmium vapor may coat the inside of the CCR. This powdery coating can easily be re-deposited if it is not properly identified and managed. Cleaning a contaminated CCR is complicated by hazardous waste issues. Short term overexposure to cadmium can produce pulmonary edema within 24 hours. Long term overexposure damages the kidneys and increases the risk of lung and prostate cancer. If masking is necessary during a high temperature experiment, both BN and GdO paints are available in the prep area of the high bay.

The double-focusing monochromaters at BT-7 (and eventually at MACS) introduce new concerns regarding Cadmium masking. Cadmium is not allowed near the sample on BT-7 or MACS because of the significantly higher neutron flux on those beam lines. The problem is that the illuminated cadmium radiates hazardous amounts of gamma particles. If a sample requires masking on these instruments, use BN or GdO paints as for high temperature experiments.

Temperature Sensor Accuracies at Your Fingertips

Have you been perusing your equipment booklets lately? Check out the new temperature sensor accuracy charts...

In the booklets found on sample environment equipment, we now provide temperature accuracies for our sensors as supplied by their respective companies. Our instruments use a variety of temperature sensors; the ones most commonly used are silicon diode DT470s, DT670s, SI410s, and an assortment of thermocouples. There is a prevailing misconception that all of the sensors have individual calibration curves; in fact, we purchase sensors based on how closely they follow a vendor's characteristic curve. A small set of possible curves is less confusing for users.

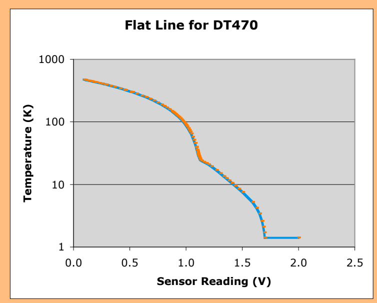
Standard Curve 10 Tolerance Bands for DT-400 Series Silicon Diodes

Type 470	2 K to 100 K	100 K to 305 K	305 K to 475 K
Band 11	±0.25 K	±0.5 K	±1.0 K

Note that temperature accuracies may drift with thermal cycling and time. This drift is greatly exacerbated when a sensor is overheated with heat guns, etc. However, it is important to remember that the greatest source of error depends on the integrity of the contact between the sensor and the sample. The sensor reflects its own temperature, and it is up to the user to ensure their sample is in good thermal contact and equilibrium with the sensor.

The best way to be assured that the controller is set up correctly and that the sensors are working properly is to check the sensor readings at room temperature **before** an experiment is started. Room temperature can now be compared to the readings found on our new digital thermometers mounted in the sample environment prep areas and labs. They have a quoted accuracy of ±0.1K. These thermometers have a second sensor on a ten-foot extension for closer comparisons.

Flat Line Sensor Curves



If you were to look at the silicon diode temperature sensor curves for our instruments, you would notice a flat line at the low temperature limit of the sensor. The sample environment team added these points for several reasons. When the controller displays a steady temperature at the sensor's calibration limit, the sensor is either at that temperature or below. Below this limit, the sensor is inaccurate, so the curve cannot be extended. However, the controller's heater will turn off if it reads a value under the sensor's low temperature limit (displayed as "T-Under"). By adding in a flat line, we avoid this scenario. If "T-Under" is ever displayed it is likely that a curve without a flat line has been accidentally selected. Look at the top of the controller or in the equipment booklet for guidance on the proper curve selection. Another motivation for the flat line is to aid the controller in properly reading temperatures; its method of interpolating near the lower limit improves when there are points extending beyond the minimum temperature.